

Porous aromatic organosilicates: surface chemistry

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Recently we have developed highly porous hybrid organosilicate materials via non-hydrolytic sol-gel methods. Silicon(IV) acetate and tris(acetoxy)silane are readily attacked by multifunctional nucleophilic building blocks such as 1,3,5-trihydroxybenzene resulting in formation of amorphous micro/mesoporous gels. As acetic acid is the only byproduct, removal of all volatiles in vacuo affords pure xerogels and the degree of condensation can thus be monitored by gravimetry. Both products have thermal stability up to 400–500 °C with BET surface areas of 990 and 933 m² g⁻¹ for silicon(IV) acetate and tris(acetoxy)silane, respectively. The product of tris(acetoxy)silane also possesses exceptionally high total pore volume of 1.36 cm³ g⁻¹. As both materials are amorphous imperfect alignment of building units limits the degree of condensation to 85–90 % and the surface therefore contains residual acetate and phenolic hydroxyl functional groups.

Herein we report a study on the chemistry of the surface groups and their reactions with alcohols, phenol and silylating reagents (Me₃SiCl, (Me₃Si)₂NH) in post-synthetic treatments. The investigation focuses on the control of surface hydrophobicity, acidity and coordination properties. Products were characterized by elemental analyses, solid-state ¹³C and ²⁹Si NMR, IR spectroscopy, nitrogen adsorption analysis, thermal analysis TG/DSC, and XRD measurements.

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